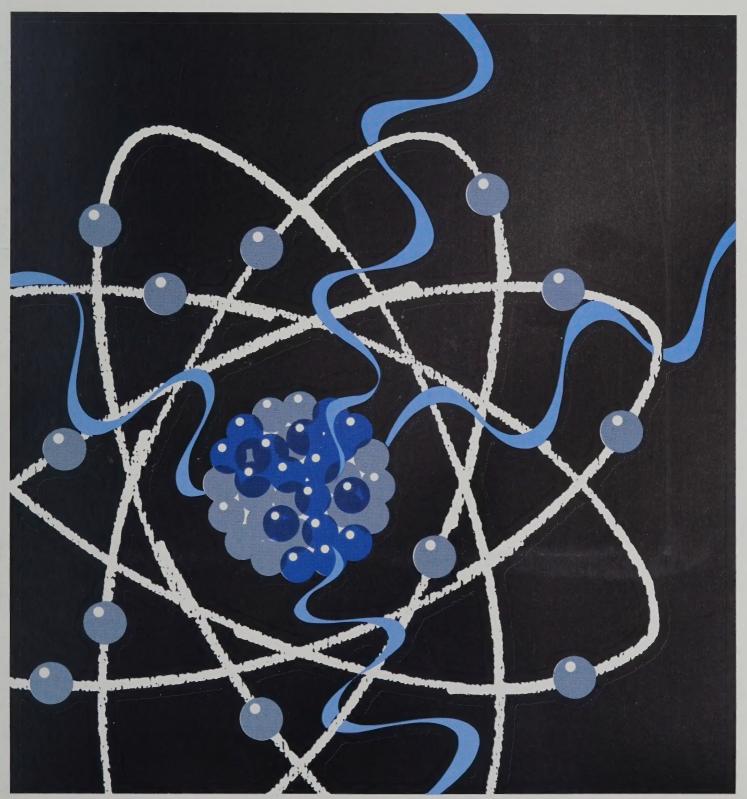
# DIVENSIONS

The magazine of the National Bureau of Standards U.S. Department of Commerce

April 1980



# COMMENT

#### MEASURING IONIZING RADIATION



Fundamental knowledge and data describing the physical and chemical effects produced by ionizing radiation in matter are essential to measure accurately ionizing radiation and to understand the effects of

ionizing radiation on living organisms.

The article on the accurate measurement of ionizing radiation using radiochromic dyes that appears in this issue of DIMENSIONS/NBS illustrates an important area of ionizing radiation research in the National Bureau of Standards' Center for Radiation Research (CRR).

The mission of the Center for Radiation Research is to provide high accuracy radiation measurement techniques and quantitative data about atomic and nuclear radiation processes. Such techniques and data are required for the safe and effective use of radiation in key technologies such as radiation therapy and diagnosis, nuclear and fusion power, industrial radiation processing, and communications. Center activities cover the electromagnetic radiation spectrum starting at the infrared, through the visible, the ultraviolet, and vacuum ultraviolet, to x rays and nuclear gamma rays.

The Center also develops measurement methods and data for key applications of electron and nuclear particle radiations, which also cover a large dynamic energy range—from a fraction of an electron volt to more than 100 million electron volts. The Center focuses its research and standards development upon the selected portions of this broad electromagnetic and particle energy continuum that are vital to the fulfillment of priority measurement needs in U.S. technology.

During the past year, CRR has directed special attention to the effects of ionizing radiation on health and safety, which have attracted a large amount of public and institutional concern. At the request of the Senate Committee on Commerce, Science, and Transportation, the Center is cooperating with the Conference of State Radiation Control Program Directors in a review of the need for an intermediate level of calibration services, such as regional facilities to better couple NBS laboratories with

State and industrial needs. This review shows clearly the need for such intermediate calibration services as well as the need for increased CRR measurement capabilities.

In its efforts to assist the States with their radiation protection measurement problems, CRR is working with the State of Illinois to set up a pilot regional calibration laboratory, with the State of Florida to set up a calibration facility for low-level survey instruments, and with the University of Michigan to test personnel dosimetry performance. Special measurements were made for the Nuclear Regulatory Commission and the Environmental Protection Agency on dosimeters used near the Three-Mile Island nuclear power plant.

Natural matrix radioactivity standards, such as human lung and liver, are being prepared to help assure accurate measurements in environmental monitoring. We are working on dosimetry measurements and standards for radiation sterilization of medical supplies, a process that replaces the use of toxic ethylene oxide. Developing methods for enhancing the extraction of information from x-ray images is part of our work on the technology for reducing patient exposure. And, because of the need for increased cooperation among the various Federal agencies concerned with ionizing radiation, the Center is represented on two recently formed interagency committees on ionizing radiation.

There is not space here to describe CRR's research and measurement activities in optical, ultraviolet, plasma, and nuclear radiation. Our goal, for all types of radiation, is to improve both the national radiation measurement base and our understanding of the interaction of radiation with the physical world.

avis & Kujatt

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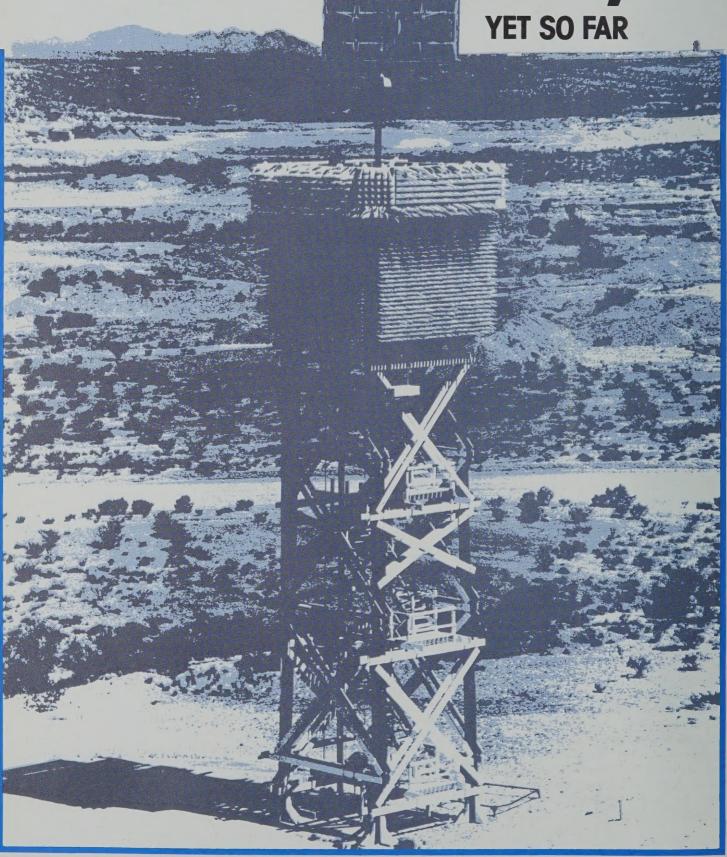
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# SO NEAR,



Two roads diverged in a wood, and

I—
I took the one less traveled by,
and that has made all the difference.

Robert Frost

The Road Not Taken

by Frederick P. McGehan

HE problem of measuring the efficiency of radio antennas was probably not on Frost's mind when he wrote those lines, but his words might well have occurred to some NBS scientists in the early 1960's when they decided to take up the radio problem. They were facing a fairly common situation in research—the project you start out with is not always the one you finish.

Originally, David Kerns and his associates at the NBS Boulder Laboratories were working on a problem of long-standing interest to physicists—how to better measure the speed of light. They were working with millimeter waves—that is, radio waves with a wavelength near one millimeter—and they planned to start out with radio waves at a very accurately known frequency and then measure the wavelength as carefully as possible. The product of frequency and wavelength would give a value for the speed of light.

On the way, they were overtaken by events. First, there was the advent of the laser. It soon became apparent that this new tool would produce a better speed-of-light measurement than could be achieved with the millimeter-wave interferometer. (And, indeed, by 1972 a team of NBS scientists had succeeded in using a stabilized laser to measure the speed of light to within about 3 parts per billion—100 times better than the best previous measurement.)

Then there were growing problems in antenna measurements. Why? An antenna does for radio waves (including microwaves) what a lens does for light—it shapes and focuses the electromagnetic energy. For some applications, such as commercial radio stations, the antenna is designed to spread the

energy as widely as possible. But for many other applications, such as the microwave relay links used for long-distance telephone, television, and data communication or for radar tracking and radio astronomy, the goal is to focus or concentrate the energy in as narrow a beam as possible so that a receiver will pick up only the energy from the desired transmitting site and no other.

One crucial measurement of an antenna is how well it concentrates the electromagnetic energy in the desired direction. The measurement parameter involved is called "gain," the ratio of the maximum beam intensity to the intensity that would exist if the same power was radiated equally in all directions. (Think of the difference between the light from a flashlight bulb hanging free in the middle of a room and the light from the same bulb in a flashlight focused on a particular point.) In a military radar system, for instance, the higher the gain of the antenna, the farther away a target can be detected.

Two other important parameters are "side lobes" and "back lobes." These represent electromagnetic energy radiated in directions other than the desired direction—energy that is wasted or can result in interference with other antenna systems and provide opportunities for unwanted eavesdropping on otherwise secure communication systems.

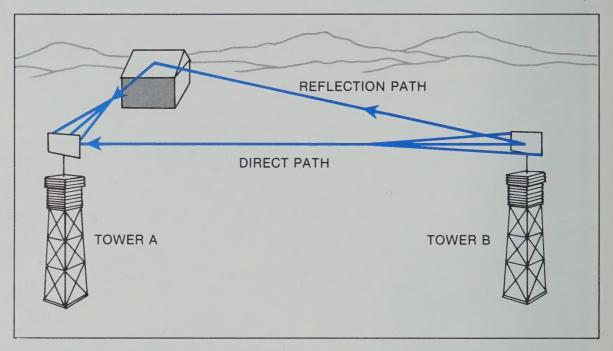
This was the early 1960's and the U.S. space program was just moving into high gear following the Soviet launch of Sputnik in 1957. Existing antenna measurement techniques provided an accuracy only within plus or minus 10 to 20 percent. Thus engineers had to overdesign antennas and systems by an equivalent amount to ensure communication between ground controllers and spacecraft. The cost to overdesign one satellite system by 10 percent was \$1 million.

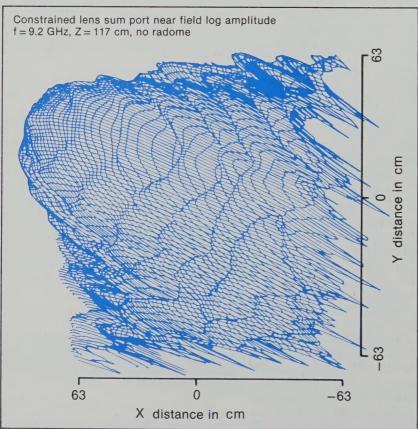
Another factor was the development of so-called "phased array antennas" at about this time. By combining many small antennas in a suitable arrangement, it was found that an arbitrary radiation pattern could be generated. Further improvements (the use of electronic phase shifters) allowed controllers to change the antenna beam direction in milliseconds in order to scan large regions rapidly and track multiple targets. The benefits for national defense and space tracking were enormous, but there was one hitch: Because of the size and complexity of these arrays, a new method had to be found for measuring them.

It was because of factors like these that this NBS team borrowed a page from Frost. They left speed-

McGehan is a writer and public information specialist in the NBS Boulder Program Information Office.

The figure below illustrates one of the problems that can be encountered in a far-field measurement situation. Some of the energy emitted by tower B is reflected off the building at the left center and picked up as reflected energy by tower A, the receiving antenna. Right is a graphic representation of the energy measured by tower A. The highest peak represents the main energy path; the smaller peaks on either side, the reflected energy.





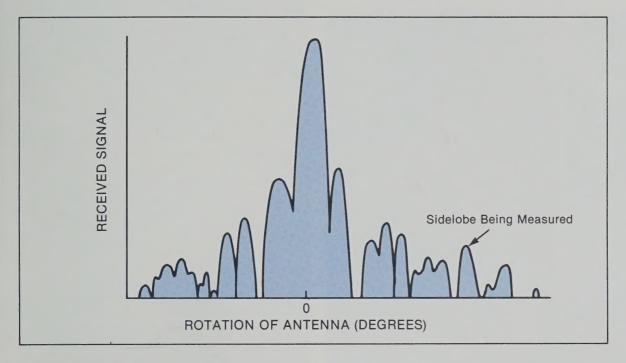
of-light experiments to the laser specialists and took the road "less traveled by"—and that has made a big difference. In fact, it produced a revolutionary way to measure antennas.

#### Far Field/Near Field

The conventional method of measuring antennas had been to use large outdoor facilities known as far-field ranges. Typically, a far-field range consists of two towers set anywhere from 300 meters to 12 000 meters apart. A transmitting antenna is placed on one tower and a receiving antenna on the other. The receiving antenna measures the beam pattern of the transmitter as it is rotated in azimuth and elevation.

The reason for this arrangement is that when electromagnetic energy is emitted from an antenna, it eventually forms a pattern whose angular shape does not change at greater distances. (That is, the basic pattern, as seen from the antenna, stays the same, although of course it expands with increasing distance.) The minimum distance at which this pattern forms is considered to be the "far-field distance" and is arrived at mathematically by squar-

This computer-generated drawing is a three-dimensional profile of the power emitted by an Air Force radar antenna as the NBS waveguide probe passed in front of it on the indoor antenna range.



ing the largest aperture dimension of the antenna, doubling that, and then dividing by the wavelength of the signal. At distances greater than this the angular shape of the beam, known as the far-field pattern, can be directly measured.

This method has provided useful measurements of antenna parameters for a number of years. But as frequencies go higher and antennas become larger, the length of the far-field range has to be increased. Because of the large amount of land required for this, some modern antennas cannot be tested on conventional far-field ranges. Other drawbacks with far-field ranges include reflections of the signal off the ground or nearby structures that result in spurious signals that cause errors in measurement. Then there are the effects of weather. Antennas have been known to blow off test stands under high wind conditions, and rain, snow, and cold temperatures can cause expensive delays in test schedules.

It was clearly desirable, therefore, to develop environmentally controlled, indoor antenna measurement facilities. The problem was how to use measurement data gathered at close range to obtain valid far-field results. At distances less than the far-field range, only a few meters from the antenna, for example, the beam is not yet formed and the field distribution is much more complicated because of a number of short range effects caused by the antenna

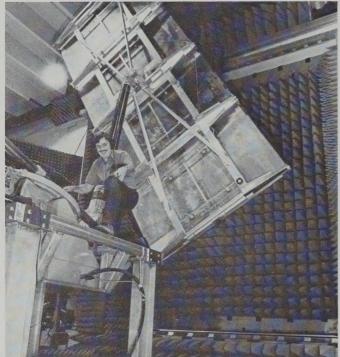
itself and the probe antenna used to measure it. So complex was the problem, in fact, that near-field antenna measurement had generally been ignored as impractical.

What changed this was that Kerns and his associates had already solved the basic theoretical problems in near-field measurements during their work on the speed of light measurement.

#### The Road Not Taken

The NBS scientists, recall, were planning to make very accurate measurements of the wavelength of microwaves. They were using a Michelson interferometer (after Albert Michelson, who used it in his experiments in the 1880's) which uses an arrangement of two synchronized beams traveling different paths to measure the wavelength of the signal. It's a very accurate method, but because of the diffraction of the energy radiating from one arm of the interferometer, the measured wavelength was longer than it would be in free space. Kerns and E. S. Dayhoff, an associate, came up with a theoretical correction for this effect.

Now correcting measured wavelength for diffraction effects is not the same problem as correcting for near-field distortions in antenna measurements, but Kerns soon realized that the same basic equations could be applied to either case. Theoretically, one could measure the field distribution



in front of the antenna and, through the use of a series of mathematical formulas, calculate the far-field characteristics of the antenna, all without going

Provided you could do the calculations. The basic mathematical operation required to obtain far-field results is called a Fourier transformation which, in this application, allows you to express a complicated radiation pattern as the sum of a number of simple plane waves. Prior to 1965, the computer time required to transform the amount of data obtained in a typical measurement would have been enormous.

Fortunately, a very efficient technique for performing the operation, known as the Fast Fourier Transform (FFT), was developed by mathematicians at Bell Laboratories and IBM's Watson Research Center. (Like many scientific advances, the FFT had its antecedents as far back as 1903.) Applied to near-field measurement theory, this FFT permits a computation that would have required an hour to be done in 3 to 4 seconds.

With the stage set by theoreticians and computer scientists, it remained for NBS physicists and engineers to put theory into practice. In 1973, a large near-field antenna measurement range was constructed at the NBS Boulder, Colo., laboratories which was based on the experience with a smaller

Douglas P. Kremer, a technician in the Electromagnetic Fields Division, rotates the large, rectangular antenna being measured in the NBS indoor measurement facility. Because the antenna was too large for conventional measurement, the bottom two-thirds was measured first and then the antenna was turned so that the top two-thirds could be measured.

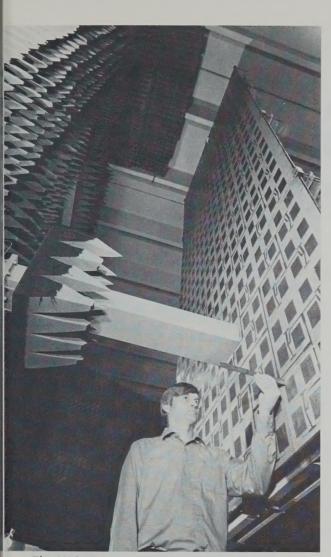
facility in use at NBS since the 1960's. Other laboratories had tried—and failed—to develop accurate, reliable near-field techniques. They failed because they did not have a rigorous theoretical foundation that included a correction for the probe antenna. NBS succeeded because it possessed the right combination of theoreticians and experimentalists as well as high-speed computing capabilities and sophisticated microwave receivers that measured phase as well as amplitude of the electromagnetic waves.

NBS also possessed a highly skilled instrument shop that was able to achieve tolerances on the order of a few thousandths of an inch. "We built the whole facility, including some equipment we already had, for \$60,000. Similar facilities are being built from scratch today for \$600,000," says Allen C. Newell, who directs the NBS near-field measurement facility.

Despite the strong lead shown by NBS, general acceptance of the near-field technique by industry had been slow until recent years. A New Mexico State University report attributes this foot-dragging to a reluctance on the part of engineers to change methods. The blockage, the report says, "is apparently a psychological one, resulting from a reluctance of engineers weaned on far-field techniques to believe that far-field patterns issuing from a computer print-out of transformed near-field data are as accurate as those obtained by direct far-field means." The report goes on to note that when the errors in near-field versus far-field measurements are compared, the errors in the near-field measurements are fewer and smaller.

There is clear evidence that the psychological barrier is falling. Hughes Aircraft Company used the design of the NBS near-field facility to construct its own indoor range in Fullerton, Calif. RCA used some of the NBS design features in a near-field facility in Moorestown, N.J., and Martin-Marietta adopted a laser interferometer technique pioneered by NBS for its near-field range in Denver, Colo.

Just as important as the facility are the computer programs that perform the theoretical calculations. There are tens of thousands of data points that have to be digested in order to measure one antenna.



panels. Significantly, the State of New Mexico is

Allen C. Newell of the Electromagnetic Fields Division uses a ruler to make an alignment adjustment to a large NASA spacecraft antenna under measurement test at NBS' indoor antenna range The checkerboard-like antenna is at the right, extending toward it is

the NBS measurement probe.

The NBS computer program has been so successful that, according to Newell, "almost everyone doing work in this area has obtained and is using our computer programs. They are the keys in almost all (near-field) measurement systems."

The New Mexico State University report compared measurements taken in the far-field (at New Mexico State's outdoor facility) with measurements taken in the near-field (at NBS) on an engineering model of a large radar antenna intended for satellite use by the National Aeronautics and Space Administration (NASA). The report, jointly authored by Keith R. Carver of New Mexico State and Newell of NBS for NASA's Johnson Space Center, concludes: "The near-field antenna test technique is intrinsically superior to far-field test techniques. when applied to measurement of antenna patterns of large planar arrays used in synthetic aperture radars." (Synthetic aperture radars are used to track extremely small objects. They are mounted on vehicles, usually airplanes, and because of the movement of the vehicle plus the way return signals are processed, give the effect of being much larger radar units.)

One obvious advantage for the near-field technique was shown when a gust of wind blew the large radar antenna off of its mounting at the New Mexico State outdoor test range and damaged the planning to construct its own near-field test range.

Another major boost for the near-field technique came in an article titled "Implementing a Near-Field Antenna Test Facility," which appeared in the September 1979 issue of Microwave Journal. Wayne A. Harmening of RCA's Government Systems Division, Moorestown, N.J., noted that a near-field test facility can be set up adjacent to a company's antenna assembly without sacrificing the test environment. "These benefits are essential in an online testing environment," Harmening wrote. His article went on to relate how RCA implemented and operates its own near-field facility at Moorestown.

Members of the NBS Electromagnetic Fields Division in Boulder have assisted in the dissemination of near-field technology by holding several courses for electrical engineers, giving numerous talks at conferences throughout the country and the world, hosting representatives of private industry who come to Boulder to learn the near-field technique, and publishing detailed information on the theory and technique involved.

Work is continuing at NBS to perfect and expand the technique. NBS scientists and engineers are developing methods that will enable the scanning of antennas on a cylindrical or spherical surface enclosing the antenna, as well as on the more familiar planar surface. This will permit a 360degree scan of an antenna, enabling the tester to tell whether the antenna is meeting performance specifications and whether there are side lobes that cannot be detected by the planar technique.

NBS scientists and engineers are examining every conceivable source of measurement error in the planar technique in order to come up with an analytical approach to error analysis. "We attacked the analytical problem and have been quite successful," Newell notes. The result has been a set of fairly simple equations that can be applied to any near-field antenna measurement to determine the estimate of error.

NBS' reputation is so strong in near-field measurements that one industry representative told Newell at a recent meeting: "Whenever a nearfield question comes up in our industry, we check with NBS first."

# NBS FY BUDGET 1981 REQUEST 981

by Mat Heyman

total of \$102,869,000 is included for the National Bureau of Standards in the Fiscal Year 1981 budget proposal sent to Congress by President Carter.

The budget request represents an increase of \$7,591,000 or 8 percent above the FY 1980 appropriation and pending pay raise supplemental. This includes \$6,049,000 for program changes and new initiatives.

In the FY 1981 request are major increases for research on very large scale integrated circuits (VLSI) and for a program begun 2 years ago to enhance the scientific competence of NBS.

Other program increases would permit NBS to meet the most urgent computing requirements of the Bureau and allow certain facilities to be modified.

"This budget request would allow us to build upon actions taken in recent years to strengthen NBS and permit Bureau resources to be used in the most responsible way in dealing with national problems," said NBS Director Ernest Ambler. "The continued funding to enhance NBS scientific competence would help ensure that the Bureau can anticipate and meet future scientific and technological needs of our Nation," Ambler noted.

A total of \$3.3 million is requested for work on very large scale integrated circuits. This proposed increase would allow NBS to develop the widely needed test procedures and measurement techniques to be used by the semiconductor industry to meet the increasing quality control demands associated with the manufacture of VLSI circuits. The NBS research effort, focused on measurement science, is expected to have a substantial impact on improving the yield of good circuits by enabling

the manufacturers to better control the numerous processes used in making VLSI circuits. In addition, the measurements and procedures developed by this effort will provide a uniform basis upon which decisions can be made to buy and sell semiconductor materials, manufacturing equipment, and completed circuits in both domestic and foreign markets.

The proposed budget includes a total of \$2 million in the area of central technical support to continue a program to enhance the scientific competence of NBS. Begun 2 years ago, this effort is directed at strengthening the technical capabilities of NBS, allowing NBS to attract top scientific talent and permitting researchers an opportunity to explore the fundamental questions that will have great practical significance in the coming decades.

Several other NBS programs would receive increases in the FY 1981 budget. Other increases include:

- A total of \$1 million for computing services. This will permit NBS to meet the most urgent current computing requirements.
- A total of \$400,000 for facilities modification. This funding will permit modifications which will give the handicapped greater access to NBS facilities in Gaithersburg and Boulder. It will also begin a long-term program to replace aging plant equipment.

One funding decrease is included in the FY 1981 budget proposal. The Field Methods Program, which includes the Experimental Technology Incentives Program, would curtail policy experiments by \$574,000.

This program will be refocused to ensure that the results of past studies are incorporated into the appropriate government policies and regulations before new studies are undertaken. The impact of this reduction will be minimized since various related activities are included in the Department's industrial innovation programs.

Heyman is chief of Media Liaison in the NBS Public Information Division.

#### **ESTIMATED NBS OPERATING FUNDS**

(Congress only)\*
(in millions of dollars)\*\*

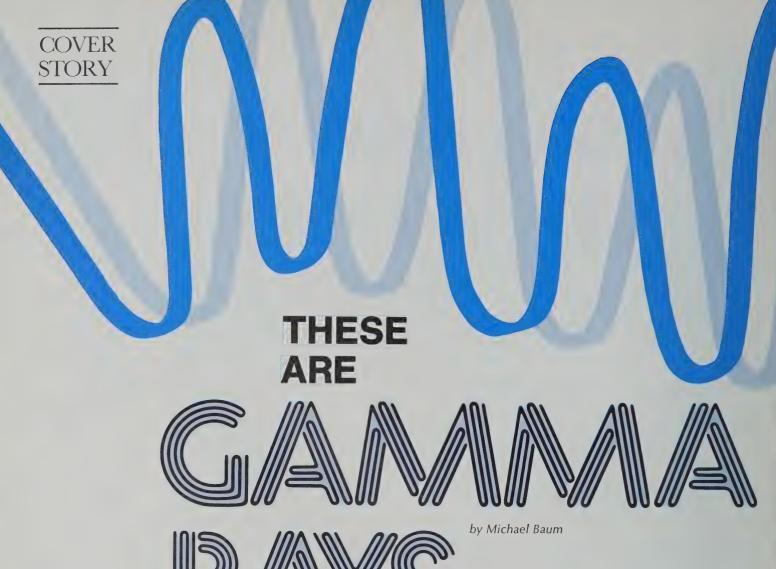
Measurement research and standards:	FY79 (actual)	FY80*** (estimate)	FY81 <sup>a</sup> (request)
Physical and chemical measurements and standards	\$17.27	16.64	17.61
Materials and thermodynamics measurements and standards	14.87	16.38	17.40
Measurement assurance program	2.12	2.53	2.68
Applied measurement program	8.44	9.22	9.73
Subtotal	42.70	44.76	47.41
Engineering measurements and standards:			
Engineering standards	2.22	1.49	1.61
Safety and performance engineering research	5.14	4.78	5.09
Technical support to industrial productivity	15.26	13.18	16.61
Mathematical science	2.80	2.82	3.01
Fire science and engineering	eren-outs	1.14	1.25
Subtotal	25.41	23.42	27.57
Computer sciences and technology	11.28	11.23	11.60
Cooperative technology	1.58	_	_
Central technical support:			
Maintenance of technical competence	4.63	8.14	11.40
Capital transfers and facilities	.20	.20	.56
Subtotal	4.83	8.34	11.97
Transfer to working capital fund	3.33	4.84	4.32
TOTAL	89.13	92.58	102.87

<sup>\*</sup>NBS receives additional funding from other Federal agencies and the private sector for research and services provided.

<sup>\*\*</sup>All figures may not add due to rounding.

<sup>\*\*\*</sup>Excludes pending pay raise supplemental of \$2.7 million.

<sup>&</sup>lt;sup>a</sup>As amended March 31, 1980.



RAYS

COLOR THEM BLUE BILL McLaughlin picks up the small vial of dye to show it off, tilting it to catch the light to best advantage. It has a subdued, rather attractive, blue-violet shade, but then pretty dyes are easy to come by. What makes this one special?

Well, this one tells you something. It's a radio-chromic dye, and the depth of the rather regal violet shade means that at some time in the past the little vial of dye absorbed a dose of 100 grays of radiation (where 1 gray = 1 joule per kilogram = 100 rads) which means that this particular dye is of more than passing interest to a wide variety of people who manufacture tires, treat cancer patients, fight insect pests, sterilize hospital equipment, and so on.

McLaughlin is a physicist in the NBS Radiation Physics Division, and as he tells the story, it began with a visitor—"had a friendly smile and a shock of white hair"—who came to his lab one day back in 1964. "He had found something unusual in his lab," McLaughlin recalled. "Ultraviolet radiation would turn these clear compounds of his colored, and he was wondering if x-rays would have the same effect."

The visitor was a private chemist, Dr. Lyman Chalkley, one of the world's authorities in dye

Michael Baum is a writer and public information specialist in the NBS Public Information Division.



photochemistry, and at that time Chalkley was developing ultraviolet sensitive dyes.

"We tried out comparatively low doses of x-rays with no discernible effect," says McLaughlin, "so I thought about higher exposures and tried it again. It turned out that it not only changed color, but it did it linearly, and gave the same dye yield at low and high dose rates."

Now when you are interested in measuring something like light or radiation, it is very handy to have an indicator that changes linearly—that is, at a constant rate—with the quantity of whatever you are measuring. On that day in 1964 there were no good chemical detectors that changed linearly at high radiation doses—upwards to 1000 grays. McLaughlin, needless to say, was immediately interested in Chalkley's dyes.

Fifteen years later, these commercially produced radiochromic dyes are in use or being investigated for industrial and civil defense dosimeters, medical radiation calibration systems, and, just possibly—anti-cancer agents.

#### Fragile Dyes

Chemical compounds that change color when irradiated are not unknown. What makes the dye systems developed by Chalkley and McLaughlin uniquely useful is a combination of qualities, including linearity, stability, sensitivity in high dose ranges, and, of course, convenience.

The basic reaction is not terribly complex, although some of the finer details are still unclear. The accompanying illustration shows a typical radiochromic dye precursor, the leucocyanide of a triphenylmethane dye based on the dye pararosaniline. (Pararosaniline has the characteristic blue-violet color. When chemically reduced by the addition of a cyanide (CN) radical, it turns clear. Leukos in Greek means white, hence a leucocyanide.)

The cloverleaf arrangement of three substituted aniline groups as substituents on acetonitrile that makes up the basic dye precursor is comparatively stable—the weakest link is the one that bonds the cyano group to it. When enough energy is added to the molecule in the form of radiation, the cyano group breaks free, the carbonium cation is formed, and the dye takes on its characteristic color. Left at that, the cyano group would quickly rebond with the dye and reverse the reaction. But if the dye is



Physicist Bill McLaughlin fills sample vials with a radiochromic dye solution. Dye has already been exposed to radiation.

in a weakly acidic environment, the cyano group will become attached to one of the hydrogen ions in the acid instead. The rest of the dye molecule will pick up a negative ion from the solution and form a colored salt in polar solution. All of this happens very quickly, in no more than a few billionths of a second.

Because the yield of the dye is linear with the radiation dose, it is comparatively simple to make dosimeters of different sensitivities, either by changing the concentration of the dye or simply by changing the thickness of the dosimeter. (Both techniques rely on the same simple principle: The more dye molecules there are to be irradiated, the more sensitively the solution changes color and the deeper the color becomes.)

The dyes can also be made in different colors. The color of the dye (after it is irradiated) depends on what chemical groups are attached to the benzene rings. (In pararosaniline, they are NH<sub>2</sub>

Radiation-initiated chemical reaction of a radiochromic dye, the leucocyanide of a triphenylmethane dye, for the parent dye, pararosaniline. This reaction is the basis for standardized radiation dosimetry using liquid or solid solutions of the dye cyanide.

groups: in hydroxyethylated pararosaniline they are  $N(C_2H_4OH)_2$  groups, and so on.) Reds, blues, greens, and yellows are available, in addition to the magenta of pararosaniline.

These dyes have one other quality, one particularly important to medical applications: The measure of dosage is nearly independent of the dose rate. It makes no difference to the radiochromic dye whether it receives comparatively low-level radiation for a long period or very intense radiation in a short burst. If the total dosage is the same, the dye will have the same reaction. It is, according to McLaughlin, the only known chemical sensor in this range with that property at dose rates up to  $10^{12}$  grays per second.

One of McLaughlin's current projects is to learn more about the basic reaction that makes radio-chromic dyes work. Using analytical techniques developed for studying very rapid reactions, he is investigating the so-called "transient species"—the formation and dissolution of chemical bonds and radical ions that may exist only for a billionth of a second during the reaction. The information gained from this work may lead to new applications for the dyes as high-resolution energy-storage devices.

#### **Tailor-Made Dosimeters**

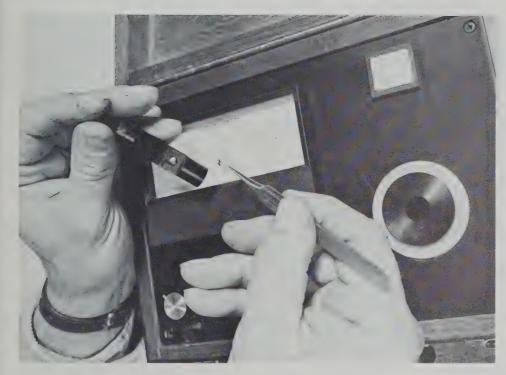
Not that there aren't sufficient applications al-

ready. Much of McLaughlin's work over the past few years in this area has been in expanding the applications for radiochromic dyes, research he has done in cooperation with the Food and Drug Administration's Bureau of Radiological Health (BRH). The BRH, among its other duties, is responsible for regulating the radiation given off by any electronic device, be it a television, microwave oven, radar unit, or, in this case, x-ray machine.

"We are trying to satisfy needs in several different areas of radiation measurement," says McLaughlin, "including industrial, medical, military, safety, and, conceivably, energy applications."

The most successful applications of radiochromic dyes have been in industry. Radiation is being used increasingly to sterilize such things as disposable plastic products for hospitals, animal feed, plastic food containers, and solid wastes. One reason for the rapid growth of this industry is the suspicion that older, chemical sterilizing agents might be carcinogenic or mutagenic.

Radiation is also being used in the plastics industry to improve certain products. The radiation causes the plastic molecules to form cross-links that add to the strength and ruggedness of the finished product. Tires, for example, are sometimes treated this way to make the elastomer easier to handle in production. The technique is also used to make



A standard industrial application of radiochromic dyes uses this portable spectrophotometer. The exposed dosimeter chip held in the tweezers is placed in the carrier and the entire assembly inserted in the instrument which measures the amount of light absorbed by the chip. The entire operation takes only a few seconds, and measurements made with this system are accurate to within about 5 percent.

dry lubricants out of waste plastics and more compact wire and cable insulation.

Another possible application for the dyes is in agriculture, where experts have been studying the use of radiation to control insect pests and extend the storage life of foodstuffs. The pest-control techniques involve irradiating male insects of nuisance species to sterilize them and then releasing the insects to mix with the breeding population. This has the advantages of leaving behind no harmful pesticides and controlling insect population without upsetting the balance of nature.

Applications like these share two characteristics—they involve fairly high doses of radiation, somewhere between 10 and 100 000 grays, and they require a certain amount of control over the dosage. Too much radiation will not only sterilize fruit flies, it will kill them. Plastics that are "overdosed" become brittle and may break in use. In such cases, the radiochromic dyes offer a cheap and convenient method of process control. In fact, dosimetry of this sort is now the method of choice for the safe processing of radiation-sterilized medical devices.

Typically, the dye is in a thin piece of plastic or gelatin about a centimeter square. This dosimeter chip is kept in a small paper envelope (remember, it's also sensitive to ultraviolet light) and irradiated along with the product. Although it's possible to

get a rough idea of the dose received by the dye simply by visually comparing the color to a chart (the darker the color, the greater the dose), readings accurate to within a few percent can be made by measuring the optical absorption of the dye with a spectrophotometer, a process that takes only a few seconds.

#### The Phantom Dye

An even more promising application for radiochromic dyes is in the measurement of medical radiation—x-ray dosimetry.

Here the problem is measuring the actual amount of energy deposited in tissues by low-energy x-ray machines used for diagnostic purposes. The problem is not as simple as it sounds. There are theoretical calculations to determine the so-called "dose distribution," but they are really good only for single energy x-ray beams and for fairly simple geometric situations.

Neither of these conditions is true for a real, live patient. For one thing, when x-rays are delivered to an area where two quite different tissues come together—the interface between muscle and bone, say, or between bone and bone marrow—the radiation scatter produces a complex dose distribution that is difficult to calculate or measure. The minute you add a radiation detector to the system to make



A more exotic application for dye dosimeters is in this specially designed dosimeter for determining complex radiation dose and spectral distributions.

a measurement, the situation gets worse, because the detector introduces its own scattering problems,

When McLaughlin and his BRH colleagues Harry Levine and Marvin Rosenstein began looking at this problem several years ago, there were two main difficulties to overcome. What was needed was a detector system that, first, had the same absorption characteristics as the tissue in the body, and second, had a high resolution—that is, the detector had to register differences in radiation dose over fairly small areas. Earlier gel bases for dye dosimeters proved too fluid—the molecules of the dye would drift about and blur the image.

They solved this problem with the invention, patented in 1977, of a plastic that was compatible with both the dye and the mildly acidic environment needed for the dosimeter. Moreover, this plastic could be cast in thin films or three-dimen-

sional shapes and formulated to mimic the radiation response of human bone and muscle tissue. This made possible the construction of radiochromic "phantoms"—imitations of bone and muscle—that could measure actual delivered doses of radiation from medical x-ray machines. Accuracy was within about 10 percent.

Much of McLaughlin's current work in dosimetry is devoted to improving this system, developing techniques of optical density measurement, and fabricating thin-film dosimeters to improve the accuracy of the measurements. In this he works with Britain's National Physical Laboratory, a counterpart of the NBS.

#### And So On

These are just some of the applications that have been found for Chalkley's clever dyes. McLaughlin has successfully used radiochromic gels to record how radiation doses given to samples in glass containers vary as the sample gets closer to the glass wall, a subject of more than passing interest to radiation chemists who use such containers for pulse radiolysis studies.

Based on McLaughlin's studies of the basic radiochromic reaction, it may be possible to develop dye dosimeters that keep a continual, real-time check on radiation levels. Since the response time of the dye is very short, one possibility, according to McLaughlin, is a detector that can monitor instantaneously very short bursts of high-energy radiation such as those from an x-ray laser or a fieldemission accelerator. No other known chemical system can do this.

In yet another area, McLaughlin is now investigating radiochromic dyes as a possible long-distance transfer standard for dosimetry—a standard of radiation measurement that can be sent from one laboratory to another to compare instrument calibration. Some 15 countries worldwide are now working with the United States to develop this idea.

Dye dosimeters might also make excellent personal radiation detectors for military, civil defense, and even industrial uses. They would be cheap, convenient, and potentially quite accurate. A private firm is now working with NBS under the Research Associate program to explore this possibility.

All in all the potential uses of radiochromic dyes seem endless, guaranteeing that in the future you will be hearing a lot more from those pretty little vials of color.

# ON LINE WITH INDUSTRY

#### NEW NBS GAS FLOW CALIBRATION SERVICE IS REFERENCED TO MASS FLOW STANDARDS

by Kent Higgins

Researchers at the National Bureau of Standards have developed a new calibration system for gas flow meters which uses a closed-loop cryogenic system to reference ambient temperature gas meters directly to liquid mass flow standards.

Before the establishment of this unique facility at the NBS Boulder, Colo., laboratories, the standard calibration method for gas flow meters, such as orifice meters, was indirect. Meters were calibrated with a liquid such as water and referenced to liquid mass flow standards. The performance of the meter for gases was predicted from those data.

The new NBS calibration system is built around the Bureau's closed-loop cryogenic flow measurement facility, which makes mass flow measurements on cyrogenic liquids. In the system, a gas such as nitrogen is passed through the flow

Higgins is a writer and public information specialist in the NBS Boulder Program Information Office.

meter to be calibrated at ambient temperatures. The nitrogen is then cooled to its liquid state and passed through the liquid mass flow reference, after which it is heated to a gas and returned to the beginning of the cycle. The closed-loop feature of the system allows the gas flow meter to be compared directly to the liquid mass flow facility.

Established in 1969, the liquid flow measurement reference system was proven over a 6-year period. Currently, this system is the flow standard for the "Cryogenic Liquid-Measuring Devices" flow measurement code which appears in NBS Handbook 44, 1979 Edition, and was approved by the 61st National Conference on Weights and Measures in 1976.

An experimental test of the new system has been performed with a 4-inch (10.16-cm) gas flow orifice meter with two different size orifice plates. The orifice meter was manufactured to the standards of the American Gas Association's Measurement Committee Report #3. These tests have confirmed that the direct comparison of gas flow measurements and mass flow standards of the same fluid are possible with the modified closed-loop system.

NBS currently is performing tests on the system with 4-inch, 3-inch (7.62-cm), and 2-inch (5.08-cm) orifice meters, each with six sets of plate sizes. Sources of systematic error in the modified system, together with the random error, also will be studied. Funding for these tests has been provided by the American Gas Association and the Gas Research Institute.

Other studies made possible with this new measurement method are 1) direct comparison of gas to liquid flow on a mass basis, 2) intercomparison of gas to water or other fluid flow on a mass basis, 3) performance of gas flow meters operating on low temperature gases, and 4) investigation of Reynolds number variations at constant mass flow.

Additional information may be obtained by writing to the NBS Thermophysical Properties Division, c/o Douglas B. Mann, Building 2, 325 Broadway, Boulder, CO 80303, or telephoning 303/499-1000, ext. 3652.

NBS staff member Clarence H. Kneebone faces the modified portion of the cryogenic flow measurement closed-loop system. Modifications shown here include water and steam heat exchangers and the main counter flow unit.



# STAFF REPORTS

### LASER-INITIATED CHEMICAL CHAIN REACTIONS

Researchers at the Joint Institute for Laboratory Astrophysics (JILA) have developed a laser-initiated technique to help understand how combustion works. The technique uses very short pulses of ultraviolet laser radiation to start specific combustion reactions in a flowing gas mixture. The complete chemical evolution of the laser-ignited reaction is monitored by observing the infrared radiation emitted by the molecules in the combustion process. Their work could provide valuable information for improving combustion technology and making engines more fuel-efficient. JILA is a joint venture of NBS and the University of Colorado.

Stephen R. Leone, Quantum Physics Division, Joint Institute for Laboratory Astro-

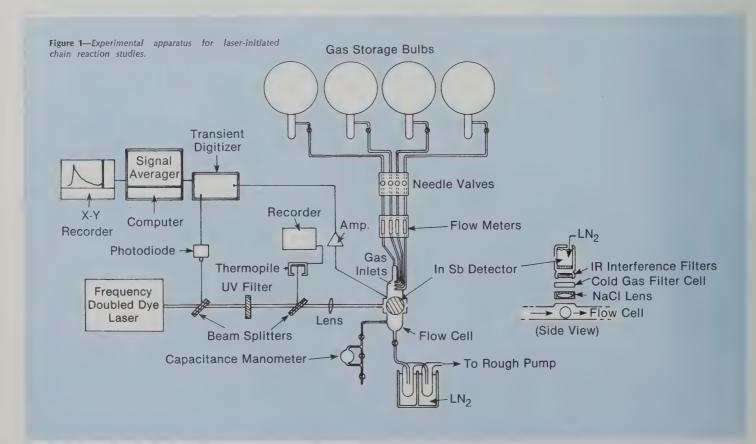
physics (JILA) and Department of Chemistry, University of Colorado, JILA A407A, 303/499-1000, ext. 3505.

A detailed understanding of chain reactions is a vital component of combustion research, accounting for the intense activity in this field in the past 60 years. Early techniques for investigating chain systems involved observing combustible gas mixtures for the onset of explosive behavior under varying temperatures and pressures. This bulk phenomenological approach misses a tremendous amount of detailed information on the actual evolution of the explosion and the transient radical species involved. With the intervening technological advances in radical detection and the development of the laser, it is now possible to study the real time behavior of chain reactions in extreme detail.

In a recent article in the Journal of

Chemical Physics 72, (1980), we\* report a detailed investigation of the experimental and mathematical analysis of real time chain kinetics initiated by radical selective laser photolysis and monitored by product vibrational chemiluminescence. The complementary techniques of laser chain ignition and state-of-theart infrared detection merge to produce a fundamentally new approach to quantitative chain analysis. By use of the pulsed laser photolysis to initiate the chain reaction, precise control of the initial radical density can be achieved. In the limit of very low densities of initial radicals. the propagation steps of the chain proceed with pseudo-first order kinetic form and detailed information on the simple chain "skeleton" can be obtained. With higher laser powers, correspondingly

\*This work was carried out in collaboration with David J. Nesbitt, Chemistry Department, University of Colorado.

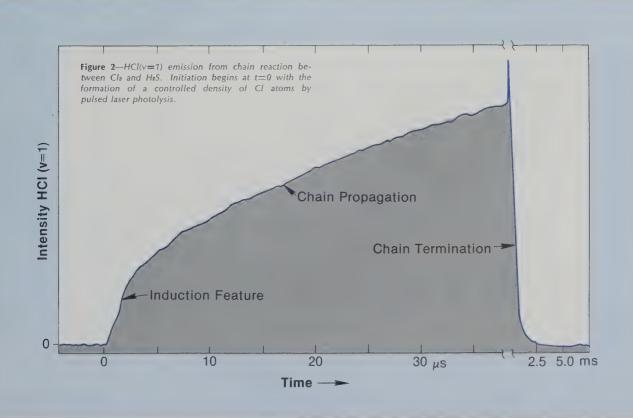


higher radical densities can be generated, enhancing the rates of radical-radical termination processes. In this manner, radical-radical reactions can be "switched on" and studied successfully.

The apparatus (figure 1) consists of a pulsed, tunable dye laser; flow reactor cell; infrared detector; and signal averaging electronics. The laser pulses generate the initial Cl atoms by dissociation of Cl<sub>2</sub>. Small fractions of the beam are used to monitor laser power and to trigger the digital electronics. The remaining laser light can be beam expanded with quartz lenses capable of varying energy density (and thus radical density) over several orders of magnitude. The reagent gases are injected into a fast flow of argon buffer gas in the reactor cell. Infrared fluorescence signals are monitored as a function of time after the laser pulse with a 77K InSb detector. Interference filters and cold gas absorption cells are used to select appropriate wavelengths of emitting species. The detector output undergoes several amplification stages and is digitized and repetitively summed in a signal averager.

A typical data run on the Cl<sub>2</sub>/H<sub>2</sub>S chain system (figure 2) displays several of the key features. The rapidly rising edge component of the emission represents the induction period, the time in which the concentrations of all radical species approach steady state. The continued quasilinear rise in the emission characterizes the steady state production of HCI(v=1)from the propagation reactions Cl +  $H_2S \rightarrow HCl (v=1) + HS, HS + Cl_2 \rightarrow$ HSCl + Cl. The precise form of this emission is actually a slowly rising exponential, due to deactivation of HCl (v=1) by reagent H<sub>2</sub>S. On very long time scales, the emission attains a maximum and then slowly decays, constituting chain termination by the radical-radical process:  $Cl + HSCl \rightarrow HCl + SCl$ . By investigating a series of such data runs with controlled variation of reagent pressures and radical densities, precise rate constant information on induction, propagation, and termination processes is obtained. The method is extremely general and can be readily applied to hundreds of chemical chain systems. As a demonstration of the versatility of the technique, several other chain systems have been investigated, including Cl<sub>2</sub>/propane, Cl<sub>2</sub>/H<sub>2</sub>, Cl<sub>2</sub>/CH<sub>3</sub> SH, Cl2/HBr, Cl2/butane, and Cl2/toluene. Each has its own characteristic behavior which is predicted precisely by the mathematical kinetics.

The laser-initiated, chain reaction technique can accommodate systems of moderate chemical complexity by forcing the kinetics into a form mathematically simple enough to yield accurate, detailed rate information. This new, versatile technique holds a great deal of potential for accu-



rate, time-resolved investigation of chemical chain reaction systems and related combustion phenomena.

# TRACE CHARACTERIZATION OF DRUGS OF ABUSE

Bureau researchers are using the Raman microprobe to identify a variety of drugs. Many of the drugs being examined—such as heroin, methadone, and cocaine—are important in forensic and toxicological studies, and the identification of very small amounts of these materials is of great importance. With the Raman technique, Bureau scientists are able to detect these drugs at concentrations significantly lower than those measured with conventional techniques.

John Blaha, Gas and Particulate Science Division, A121 Chemistry Building, 301/ 921-2872.

The ability to detect trace amounts of organic materials is of great interest especially in the areas of forensic or toxicological analysis. A wide variety of techniques is currently employed in the trace analysis of organic compounds. There remains a continuing demand for increased sensitivity and selectivity in the determination and identification of these materials.

Molecular characterization of microscopic samples by Raman spectroscopy has been previously described in *DIMENSIONS/NBS*.\* Recently the Raman microprobe has been used to identify the presence of a number of addictive drugs (heroin, morphine, and cocaine, for example) in micrometer-size particles whose masses are estimated to be 1-10 picograms. Initial studies have been directed to the characterization of individual microparticles of these materials.

Powder samples dispersed on sapphire substrates were examined in the NBS Raman microprobe with no other sample handling or preparation. Raman spectra were obtained by using the 514.5 nm line of an argon/krypton ion laser for excitation of the sample.

Figures 1 and 2 show the Raman spectra obtained from single microparticles of morphine sulfate and heroin (diacetyl

\* The May 1976 issue of DIMENSIONS carried a report on the development at NBS of a unique laser-Raman microprobe designed to enable the spectroscopic analysis of microsamples, and of microparticles in particular. In this new instrument, single particlessupported by a suitable substrate—are moved into the focal point of a laser beam (utilizing any one of several "excitation" frequencies in the visible region of the spectrum) and the light scattered by the sample is analyzed for its spectral content. The scattered light contains the Raman spectrum of the particle which is diagnostic of molecular and crystal vibrations in the solid. Particles, as other microscopic samples or sample regions, composed of a broad range of inorganic compounds, organic solids, and polymers, show the normal Raman effect and the principal molecular constitutents can therefore be identified. The application of Raman spectroscopy to single particle analysis thereby offers the opportunity for the chemical speciation of major elements and provides a new insight into the microscopic domain unattainable by other microprobes. The new instrument and technique have been applied to the investigation of diverse problems in microanalysis.

morphine), respectively. The spectra are complex as can be observed in the spectral region between 200 and 1600 cm<sup>-1</sup>.

A comparison of these spectral band patterns and their intensities shows distinct differences that allow an unambiguous identification of these closely related materials. Especially evident in a comparison are the greater number and intensity of bands in the C-H stretching region near 3000 cm<sup>-1</sup> in the spectrum of heroin.

Another class of drugs that has been analyzed is the barbiturates. Figure 3 presents the Raman spectrum obtained from a particle of secobarbitol (secanol). All of the materials examined could be identified by their characteristic Raman band patterns. This is the first time that these materials have been identified in the picogram mass range.

Raman spectra, such as those presented, are diagnostic of the components of the sample. Characterization of the individual particles in a powder sample is possible at extremely low levels. Investigations are being conducted to determine the potential of the Raman technique in the characterization of multicomponent fractions, separated by chromatographic methods, in a static mode where a portion of the effluent is trapped and the solid analyte is isolated on a Raman substrate for measurement in the microprobe.

18 DIMENSIONS/NBS

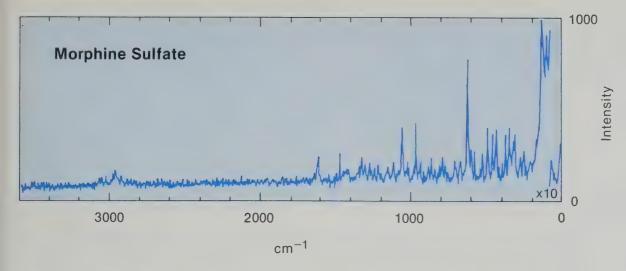


Figure 1—Raman spectrum obtained from a  $\sim$  5 X 6  $\mu m$  particle of morphine sulfate.

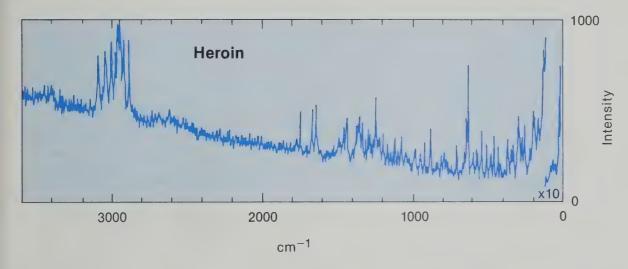


Figure 2—Raman spectrum obtained from a  $\sim$  4 X 4  $\mu$ m particle of heroin.

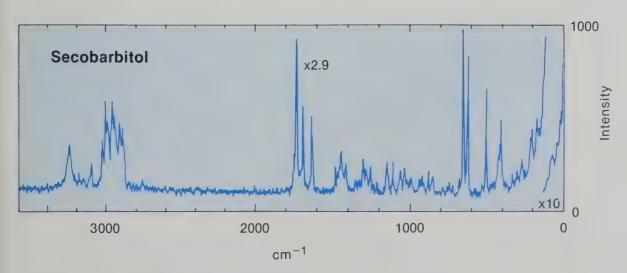


Figure 3—Raman spectrum obtained from a  $\sim$  5 X 8  $\mu$ m particle of secobarbitol.

# CONFERENCES

For general information on NBS conferences, contact JoAnn Lorden, NBS Public Information Division, Washington, D.C. 20234, 301/921-2721.

# CONFERENCE ON PRECISION MEASUREMENT AND FUNDAMENTAL CONSTANTS

The National Bureau of Standards will host the Second International Conference on Precision Measurement and Fundamental Constants at its headquarters in Gaithersburg, Maryland, from June 8 to 12, 1981.

Conference organizers hope to provide an international forum for scientists actively engaged in experimental and theoretical research on precision measurements relating to fundamental physical constants and to the testing of related theory. The last such meeting was held at NBS in August of 1970.

One goal of the 1981 Conference will be to gather additional data for the 1981 adjustment to the values for fundamental constants recommended for international use. This adjustment (using the least-squares method) is being carried out by the Task Group on Fundamental Constants of the Committee on Data for Science and Technology (CODATA).

Proposals for papers for the conference are now being solicited. Topics of interest include the absolute realization of basic measurement units; measurements of fundamental atomic constants such as the Rydberg, the fine-structure, and the gravitational constants; and high-precision tests of quantum electrodynamics and similar fundamental theories. Emphasis is placed on assessment of the present state of precision measurement, basic limitations, and possible future avenues for advances.

Those interested in attending the 1981 Conference or who need more information should contact Dr. B. N. Taylor, B258 Metrology Building, NBS, 301/921-2701.

# SEMINAR ON CERAMICS AS ARCHAEOLOGICAL MATERIAL

The Seminar on Ceramics as Archaeological Material will be held on Monday, September 29, 1980, in the Green Auditorium of the National Bureau of Standards and on Tuesday, September 30, 1980, in the Baird Auditorium of the Natural History Building at the Smithsonian Institution. If the meeting continues through Wednesday, October 1, 1980, the sessions will be held in the Carmichael Theater in the History and Technology Building of the Smithsonian Institution.

This seminar is one of a continuing series, held jointly by the Conservation Analytical Laboratory of the Smithsonian Institution and the National Measurement Laboratory of NBS, on the general subject of the application of materials and measurement sciences to archaeology and museum conservation.

The purpose of this seminar is to further the application of the tools of the materials and measurement sciences to archaeological problems involving ancient ceramics, and to do this by providing a forum for thoughtful discussion and exchange of views and experiences. The seminar will be composed of two elements: oral presentation of major papers and a poster presentation of papers describing specific and detailed research results. In the papers, the authors should develop as much as possible both the material science approach and the archaeological problem to which it is applied. Indeed, it is one of the aims of the seminar to bring together in the discussion both archaeologists and physical scientists with mutual interests in archaeological ceramics.

Areas of discussion will include: physical science studies whose ultimate goal is to reveal information on the ancient ceramics; studies of composition, both

chemical and mineralogical, for investigation of provenience, sources, and trade patterns; and, perhaps, standardization of physical and chemical measurements and the use of statistical and multivariate techniques in the analysis of data.

It is planned to publish the Proceedings through the Smithsonian Press. Both oral and poster session papers will be eligible for inclusion, but manuscripts must be submitted at the seminar to facilitate publication.

Those wishing to present papers should notify Alan D. Franklin, A329 Materials Building, NBS, Washington, DC 20234 (301/921-2901), by July 1, 1980. The information provided should include: authors' names and addresses (underline the speaker's name); title; whether paper is intended for poster or oral presentation, and if the latter, maximum and minimum speaking times; and a brief abstract.

For further information concerning the seminar, contact Jacqueline Olin, Conservation Analytical Laboratory, Smithsonian Institution, Washington, DC 20560 (202/381-5714).

#### CONFERENCE CALENDAR

#### May 1-2

IMPLANT RETRIEVAL: MATERIAL AND BIOLOGICAL ANALYSIS, NBS, Gaithersburg, MD; sponsored by NBS, DOC, FDA, DHEW, VA, and ASTM; contact: A. W. Ruff, B118 Materials Building, 301/921-2966.

#### May 5-7

TOPICAL CONFERENCE ON BASIC OPTICAL PROPERTIES OF MATERIALS, NBS, Gaithersburg, MD; sponsored by NBS in cooperation with OSA; contact: Albert Feldman, A251 Materials Building, 301/921-2840.

#### May 8-9

TRACEABILITY FOR IONIZING RADIATION MEASUREMENTS, NBS, Gaithersburg, MD; sponsored by NBS; contact: H. T. Heaton, C229 Radiation Physics Building, 301/921-2551.

#### May 13-15

METROLOGY OF MODERN ELECTRONIC INSTRUMENTATION, NBS, Gaithersburg, MD; sponsored by NBS; contact: Barry A. Bell, B162 Metrology Building, 301/921-2727.

#### May 13-15

MEDILOG 80, NBS, Gaithersburg, MD; sponsored by NBS and DOD; contact: Charles Hulick, A740 Administration Building, 301/921-3465.

#### **May 29**

IEEE COMPUTER CONFERENCE, NBS, Gaithersburg, MD; sponsored by NBS and IEEE; contact: Frances Nielsen, B212 Technology Building, 301/921-2601.

#### **June 2-6**

6TH INTERNATIONAL CONFERENCE ON VACUUM ULTRAVIOLET RADIATION PHYSICS, UNIVERSITY OF VIRGINIA, CHARLOTTESVILLE, VA; sponsored by NBS, NRL, University of Virginia, NSF, DOE, IUPAP; contact: Robert Madden, A251 Physics Building, 301/921-2611.

#### **June 2-4**

FIFTH INTERNATIONAL SYMPOSIUM ON ULTRASONIC IMAGING AND TISSUE CHARACTERIZATION, NBS, Gaithersburg, MD; contact: Melvin Linzer, A366 Materials Building, 301/921-2611.

#### **June 4-6**

SECOND INTERNATIONAL SYMPOSIUM ON ULTRASONIC MATERIALS CHARACTERIZATION, NBS, Gaithersburg, MD; sponsored by NBS and ASNT; contact: Harold Berger, B312 Physics Building, 301/921-3331.

#### June 19

19TH ANNUAL TECHNICAL SYMPOSIUM PATHWAYS TO SYSTEM INTEGRITY, NBS, Gaithersburg, MD; sponsored by NBS and ACM; contact: Carol Wilson, A252 Technology Building, 301/921-3861.

#### June 22-27

NATIONAL CONFERENCE ON WEIGHTS AND MEASURES, Shoreham-Americana, Washington, DC; sponsored by NBS and NCWM; contact: Harold Wollin, A211 Metrology Building, 301/921-3677.

#### June 23-25

HIGH RESOLUTION INFRARED APPLICATIONS AND DEVELOPMENTS, NBS, Gaithersburg, MD; sponsored by NBS; contact: Jon Hougen, B268 Physics Building, 301/921-2021.

#### July 22-24

NBS EMI METROLOGY SEMINAR, NBS, Gaithersburg, MD; sponsored by NBS; contact: M. Gerald Arthur, EMI/Radiation Hazards Group, Electromagnetic Fields Division, NBS, Boulder, CO, 303/499-1000, ext. 3603.

#### **August 14-15**

ASME SYMPOSIUM ON CRITICAL MATERIALS AND FABRICATION ISSUES, St. Francis Hotel, San Francisco, CA; sponsored by NBS and ASME; contact: Jeffery Fong, A302 Administration Building, 301/921-2631.

#### September 29-October 1

CERAMICS AS ARCHAEOLOGICAL MATERIAL, NBS, Gaithersburg, MD; sponsored by NBS and Smithsonian Institution; contact: Alan Franklin, A355 Materials Building, 301/921-2901.

#### October 7-9

COAL CONVERSION, NBS, Gaithersburg, MD; sponsored by NBS and DOE; contact: Samuel Schneider, B308 Materials Building, 301/921-2894.

# PUBLICATIONS

## NBS REVIEWS SELECTED NDE TESTS FOR INTEGRATED CIRCUITS

Harman, G. G., Nondestructive Tests Used to Insure the Integrity of Semiconductor Devices with Emphasis on Acoustic Emission Techniques, Nat. Bur. Stand. (U.S.), Spec. Publ. 400-59, 72 pages (Sept. 1979) Stock No. 003-003-02116-4, \$3.50.\*

Nondestructive tests used to check the mechanical integrity of semiconductor devices are reviewed in a new publication by the National Bureau of Standards' Center for Electronics and Electrical Engineering. The publication features a review of the uses of acoustic emission tests for semiconductor devices and hybrid microcircuits.

The need for highly reliable semiconductor devices, particularly in military and space applications, has led to the widespread application of a number of non-destructive evaluation (NDE) methods for checking properties such as the strength of wire bonds in the circuit, the integrity of the seal in packaged devices, and the presence of loose particles inside packaged devices.

The tests vary in usefulness and accuracy, and many involve a necessary element of human judgment. Most of them are described in the military testing standard MIL-STD-883.

The first part of this NBS Special Publication is concerned with an overview of six of these methods: the nondestructive wire bond pull test, internal visual inspection, temperature cycling and shock, package seal integrity, burn-in (to remove early failures), and particle impact noise detection.

The second section includes an introduction to the theory of acoustic emission testing with emphasis on its application to the semiconductor industry. There is an overview of published papers on the subject, and a look at how an acoustic emission technique devised at NBS can be applied to the mechanical testing of beam lead, flip chip, and tape bonded integrated circuits as well as the bonds by which components are attached to the substrate in modern hybrid circuits.

# ADVICE FOR STANDARDS WRITERS, REGULATORS

Harter, P. J., Regulatory Use of Standards: The Implications for Standards Writers, NBS GCR 79-171, for sale by the National Technical Information Service, Springfield, VA 22161, \$11.00; order by #PB 80-120579.

Ways in which private standards developers and government regulators can perform more effectively through improved mutual understanding of each other's roles and needs are discussed in Regulatory Use of Standards: The Implications for Standards Writers, the report of a special study conducted for the National Bureau of Standards. The report was prepared by Philip J. Harter, an attorney with extensive background in administrative law and government regulation who has served in several government posts.

The 286-page document presents material designed to help standards-writing organizations prepare standards that are acceptable for use in regulations or as an alternative to regulation. It also provides Harter's personal suggestions about how regulatory agencies might improve their relationships with private sector organizations. The report is limited solely to the regulatory use of standards developed under the auspices of a non-governmental organization. In releasing the report, Dr. Lawrence Eicher, Director of the NBS Office of Engineering Standards, said that the study "deserves the attention of both

the standards community and the regulatory agencies" that would like to "better tap the expertise represented by the private sector."

Harter's study details: use of standards in the regulatory process; administrative law and procedures; balance and due process; methods of preparing standards so as to facilitate agency review; the legal resolution of technical issues; achievement of "full consensus" in theory and in practice; performance versus design standards; antitrust considerations; applications of the Federal Advisory Committee Act; and testing procedures.

In the concluding part of the study, Harter focuses on complaints of standards writers about agencies and recommends agency actions that would improve the relationship.

#### SECOND SUPPLEMENT FOR LNG MATERIALS AND FLUID USER'S MANUAL

To order, make check or money order payable to NBS and send to LNG Materials and Fluids User's Manual, Thermophysical Properties Division, NBS, Boulder, CO 80303. For additional information, write the Thermophysical Properties Division or telephone 303/499-1000, ext. 4108. Cost of second supplement is \$15.

The National Bureau of Standards has published the second supplement of the LNG (liquefied natural gas) Materials and Fluids User's Manual. The supplement contains expanded and updated LNG data either generated experimentally by NBS or assessed and evaluated by NBS.

The supplement, which consists of 77 graphs and three wall charts, includes data

<sup>\*</sup>Publications cited from this point on may be purchased at the listed price from the U.S. Government Printing Office, Washington, D.C. 20402 (foreign: add 25%). Microfiche copies are available from the National Technical Information Service, Springfield, VA 22161. For more complete periodic listings of all scientific papers and articles produced by NBS staff, write: Editor, Publications Newsletter, Administration Building, National Bureau of Standards, Washington, D.C. 20214.

#### OF THE NATIONAL BUREAU OF STANDARDS

for aluminum alloys, stainless steels, concrete, materials specifications for concrete, thermophysical properties of propane, and data for the methane-propane system. The two color graphs express values in customary (inch-pound) and SI (metric) units. The wall charts are for pure propane thermophysical data (enthalpy-entropy, pressure-enthalpy, and temperature-entropy).

A limited number of the first edition of the *LNG Materials and Fluids User's Manual* and expandable binder are available at \$35. The first edition consists of 138 graphs and 6 wall charts, containing data on pure methane and nitrogen; binary mixtures of methane and nitrogen; and elastic, thermal, and mechanical properties of aluminum alloys (3004, 5085, and 6061), stainless steels (304, 305L, 310, and 316), nickel steels (2.25, 3.5, 5, and 9 percent alloyed), and invar. The wall charts are for pure methane and nitrogen thermophysical data.

The first supplement (cost: \$15) contains 95 graphs and 3 wall charts with data for: aluminum alloys, nickel steels, and stainless steels; thermal insulations (polystyrene, polyurethane, and polyvinyl chloride foams, balsa, perlite, cellular glass, and glass fiber); materials specification tables; thermophysical properties of ethane; and binary and multicomponent mixtures of LNG components. The wall charts are for pure ethane thermophysical data.

The intent of the manual is to provide in graphic form complete property data and related information for the effective generation, transport, and use of LNG. The research has been sponsored by the American Gas Association, Inc., the Gas Research Institute, the Department of Commerce's Maritime Administration, the American Bureau of Shipping, and the NBS Office of Standard Reference Data.

#### PUBLICATIONS LISTING

#### **Analytical Chemistry**

Myklebust, R. L., Fiori, C. E., and Heinrich, K. F. J., Frame C: A Compact Procedure for Quantitative Energy-Dispersive Electron Probe X-ray Analysis, Nat. Bur. Stand. (U.S.), Tech. Note 1106, 111 pages (Sept. 1979) Stock No. 003-003-02113-0, \$4.00.

#### **Building Technology**

Kovacs, W. D., and Yokel, F. Y., Soil and Rock Anchors for Mobile Homes—A State-of-the-Art Report, Nat. Bur. Stand. (U.S.), Bldg. Sci. Ser. 107, 164 pages (Oct. 1979) Stock No. 003-003-02121-1, \$4.75.

#### **Computer Science and Technology**

Guideline for Selection of Data Entry Equipment, Nat. Bur. Stand. (U.S.), Fed. Info. Process. Stand. Publ. (FIPS PUB) 67, 23 pages (Sept. 1979).

#### **Health and Safety**

Adler, S. C., and Pierman, B. C., A History of Walkway Slip-Resistance Research at the National Bureau of Standards, Nat. Bur. Stand. (U.S.), Spec. Publ. 565, 36 pages (Dec. 1979) Stock No. 003-003-02142-3, \$1.75.

Kramer, J. J., Ed., The Role of Behavioral Science in Physical Security. Proceedings of the Third Annual Symposium, May 2-4, 1978, Nat. Bur. Stand. (U.S.), Spec. Publ. 480-38, 110 pages (Dec. 1979) Stock No. 003-003-02149-1, \$4.25.

#### **Electronic Technology**

Ham, W. E., Semiconductor Measurement Technology: Comprehensive Test Pattern and Approach for Characterizing SOS Technology, Nat. Bur. Stand. (U.S.), Spec. Publ. 400-56, 370 pages (Dec. 1979) Stock No. 003-003-02144-6, \$8.00.

Harman, G. G., Semiconductor Measurement Technology: Nondestructive Tests Used to Insure the Integrity of Semiconductor Devices, with Emphasis on Acoustic Emission Techniques, Nat. Bur. Stand. (U.S.), Spec. Publ. 400-59, 72 pages (Sept. 1979) Stock No. 003-003-02116-4, \$3.50.

#### Engineering, Product and Information Standards

Wollin, H. F., Ed., Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices, Nat. Bur. Stand. (U.S.), Handb. 44, 1979 Edition, 213 pages (Dec. 1979) Stock No. 003-003-02143-1, \$6.00.

#### Fire Research

Sherald, M. A., Ed., Fire Research and Safety. Proceedings of the Third Joint Panel Conference of the U.S.—Japan Cooperative Program in Natural Resources held at the National Bureau of Standards, Gaithersburg, MD, Mar. 13-17, 1978, Nat. Bur. Stand. (U.S.), Spec. Publ. 540, 729 pages (Nov. 1979) Stock No. 003-003-02141-5. \$8.00.

#### Measurement Science and Technology Policy and State-of-the-Art Surveys

Marshall, H. E., and Ruegg, R. T., Efficient Allocation of Research Funds: Economic Evaluation Methods with Case Studies in Building Technology, Nat. Bur. Stand. (U.S.), Spec. Publ. 558, 56 pages (Dec. 1979), Stock No. 003-003-02132-6, \$2.50.

Steiner, B. W., An Institutional Plan for Developing National Standards with Special Reference to Environment, Safety, and Health, Nat. Bur. Stand. (U.S.), Monogr. 165, 24 pages (Sept. 1979) Stock No. 003-003-02094-0, \$4,00.

#### Metrology: Physical Measurements

McAdam, D. L., Ed., Contributions to Color Science, by Judd, D. B., Nat. Bur. Stand. (U.S.), Spec. Publ. 545, 760 pages (Sept. 1979) Stock No. 003-003-02126-1, \$14.00.

Kamas, G., and Howe, S. L., Eds., Time and Frequency Users' Manual, Nat. Bur. Stand. (U.S.), Spec. Publ. 559, 256 pages (Nov. 1979) Stock No. 003-003-02137-1, \$6.00.

#### Standard Reference Data

Goldberg, R. N., Evaluated Activity and Osmotic Coefficients for Aqueous Solutions: Bi-Univalent Compounds of Lead, Copper, Manganese, and Uranium, J. Phys. Chem. Ref. Data 8, No. 4, 1005-1050 (1979).

Goldberg, R. N., Nuttall, R. L., and Staples, B. R., Evaluated Activity and Osmotic Coefficients for Aqueous Solutions: Iron Chloride and the Bi-Univalent Compounds of Nickel and Cobalt, J. Phys. Chem. Ref. Data 8, No. 4, 923-1004 (1979).

Lovas, F. J., Lutz, H., and Dreizler, H., Microwave Spectra of Molecules of Astrophysical Interest. XVII. Dimethyl Ether, J. Phys. Chem. Ref. Data 8, No. 4, 1051-1108 (1979).

Corliss, C., and Sugar, J., Energy Levels of Potassium, K I through K XIX, J. Phys. Chem. Ref. Data 8, No. 4, 1109-1146 (1979).

Matula, R. A., Electrical Resistivity of Copper, Gold, Palladium, and Silver, J. Phys. Chem. Ref. Data 8, No. 4, 1147-1298 (1979).

#### Other Subjects of General Interest

Ruler: 15 cm/6 in, with Metric-Customary Units and Equivalent, Nat. Bur. Stand. (U.S.), Spec. Publ. 376, Both sides (Re-issued Jan. 1979) Stock No. 003-003-01080-4, 60 cents; or \$9.90 per 100.

# NEWS BRIEFS

STRUCTURE STUDIES OF POLYMER IMPLANT MATERIALS. NBS scientists have recently initiated a program with the Food and Drug Administration's Bureau of Medical Devices (BMD) to develop measurement techniques and obtain data on the long-term mechanical performance of ultrahigh molecular weight polyethylene. Current trends to implant prosthethic devices made of this kind of polyethylene in younger, more active patients requires a better understanding of how structural features of high polymers affect mechanical properties and long-term performance. Data gathered through the NBS/BMD program should help assure the reliability of implant devices and optimize the processing techniques used by medical manufacturers.

TEST CRACKS FOR LIQUID PENETRANTS. A Standard Reference Material (SRM) to be used in quality testing of liquid penetrant inspection systems is now under development at NBS. Such systems are widely used by metal parts manufacturers for detection of minute surface openings such as cracks, porosity, seams, and folds. When research and certification are completed the SRM will consist of a small block of nickel containing a series of carefully characterized surface cracks of certified size. The availability of these standards will help manufacturers establish the detection sensitivity of their liquid penetrant systems and assure that contamination and other environmental effects are not decreasing the quality of their measurements.

TESTS FOR WATER HEATER-HEAT PUMPS. Work is under way within the NBS Center for Consumer Product Technology to develop a test procedure for combination water heater-heat pumps so that meaningful comparisons between this type of unit and conventional water heaters can be made. This device is dedicated to the generation of domestic hot water using the existing hot water storage tanks found in most households. The heat pump replaces any other energy source (gas, oil, or electricity) and is designed to be a retrofit device which can be installed by the homeowner.

ENERGY MODELS. NBS Special Publication 569, Validation and Assessment Issues of Energy Models, contains the complete proceedings of the workshop on that topic held in January 1979. Topics include both theoretical and applied use of large scale models, modeling and its relation to policy, and problems in model assessment. Order from U.S. Government Printing Office, Washington, DC 20402, Stock No. 003-003-02155-5. Price \$9.50.

RESEARCH NBS ANALYZES EVALUATION METHODS. INVESTMENT: Alternative methods for evaluating research investments are reviewed in a new publication by the NBS Center for Building Technology (CBT). The study is designed to assist in the efficient allocation of limited funds by research organizations. Net benefits and rate-of-return methodologies are applied to two case studies involving CBT: heavier asphalt shingle for roofing and reduced size venting in plumbing. Both studies showed high rewards: about \$4 billion in shingle savings (net benefits) from 1962-1964, and an estimated \$105 million savings in venting costs from 1975-1985. Single copies of Efficient Allocation of Research Funds: Economic Evaluation Methods with Case Studies in Building Technology, Spec. Publ. 558, are available for \$2.50 from U.S. Government Printing Office, Washington, DC 20402, Stock No. 003-003-02132-6.

#### **NEXT MONTH IN**

# DIVENSIONS



Plastic manufacturers must prove that materials used in food packaging do not contaminate the food they are supposed to protect. Right now the process of developing and testing a new plastic packaging material could take from 5-10 years and cost \$200,000 to \$2 million. In the next issue of DIMENSIONS read about NBS efforts to facilitate this testing through the development of predictive models.

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